5.1.5. Gravimetric Standards

New Blending Manifold

An ultraclean gas blending manifold was designed and built for the preparation of gravimetric standards. The main manifold system (**Figure 5.16**) was made using electropolished stainless steel tubing (6.35 mm o.d.) that was connected together using an automatic tube welder providing relatively smooth weld joints on the interior surface of the tubing. Welded, metal-gasket face-seal fittings were also used to connect the subsections of the manifold together and at the cylinder connection ports. Packless all-metal diaphragm valves with welded, metal-gasket face-seal fittings were used at all valve locations. Three strain gauges were installed on the manifold system for pressure measurements. A 0-20.7 MPa gauge is used for measuring pressure in the main part of the manifold. Two additional gauges measure pressures from vacuum to 103.4 kPa and from vacuum to 1.33 kPa. These low pressure gauges are isolated from the main part of the manifold by a valve in the vacuum segment of the manifold. The low pressure gauges are protected from high pressures by using a high-vacuum solenoid valve wired to a pressure-readout meter. Vacuum is achieved using a high flow rate vacuum pump. A foreline trap containing molecular sieve prevents oil vapors from entering the manifold. The manifold can be evacuated to approximately 4 Pa. A gas purification section of the manifold (Figure 5.17) is used for further purification of diluent gases such as air or N_2 supplied by commercial gas suppliers. Two traps containing Ambersorb and Molecular Sieve 13X can be heated to 250°C while they are purged with high-purity N_2. The traps are then cooled to room temperature prior to use. The N_2 used to purge the traps can also be used to purge the manifold of residual gases.

Standards

A suite of calibration standards containing N_2O and CO_2 in air were prepared this year for the CSIRO Division of Atmospheric Research. The standards were gravimetrically prepared at nominal mixing ratios ranging from 265 ppb to 345 ppb of N_2O. Carbon dioxide was added to the parent N_2O mixture that was used to prepare the ppb level N_2O mixtures at nominal mixing ratios ranging from 330 ppm to 430 ppm.Standards containing methyl bromide (CH_3Br) and methyl chloride (CH_3Cl) in air were prepared this year. In the past, both methyl halides were used as refrigerants, solvents, and in organic synthesis [*Braker and Mossman*, 1980]. Methyl bromide is mostly used as a pesticide. Small aliquots of each compound were added to individual Aculife-treated aluminum compressed-gas cylinders providing ppb level mixtures. The Aculife treatment provides an internally inert surface that prevents both methyl halides from reacting with the aluminum. The mixtures were then blended together into one cylinder. Gravimetric standards containing from 5 to 25 ppt of CH_3Br and 400 to 1000 ppt of CH_3Cl were then prepared. The response factors (GC-MS response per mole injected) for both ppb and ppt level CH_3Br and CH_3Cl standards were consistent to within 5%.

Standards containing HCFC-141b and HCFC-142b were also prepared using the same gravimetric techniques used to prepare the methyl halide standards. Single component standards containing each HCFC were prepared at ppb levels. The standards at ppb levels were combined together, and the mixture containing both compounds was used to prepare standards at 5, 25, 50, and 500 ppt with a ratio of approximately 1:1 for HCFC-141b and HCFC-142b. Response factors determined by GC-MS analysis of each HCFC within the suite of standards (ppt to ppb levels) were consistent to within 5%.

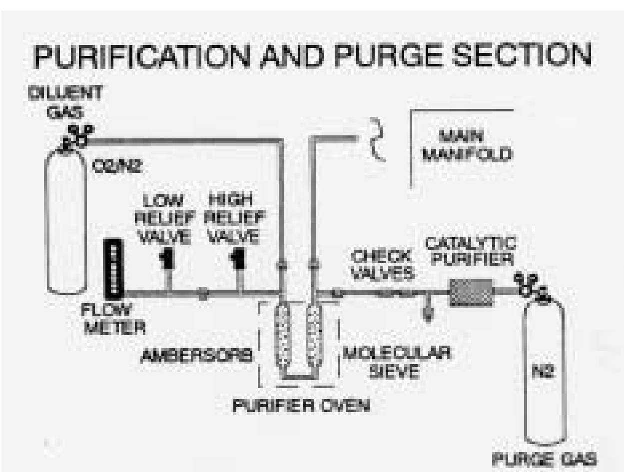

Fig. 5.16. Main section of gas blending manifold.

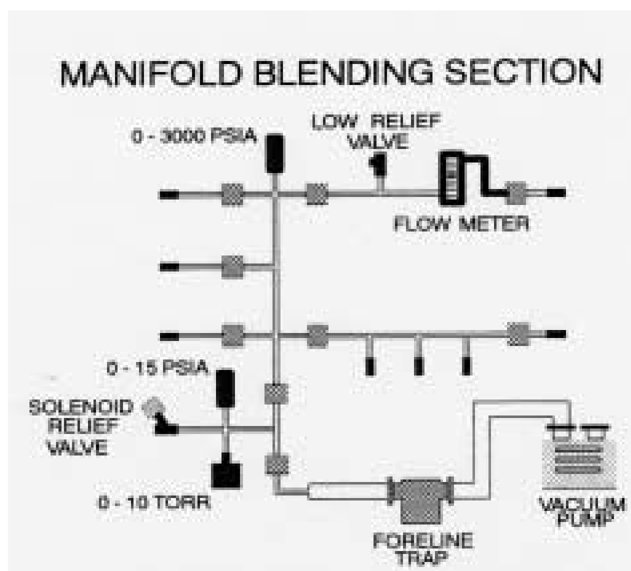

Fig. 5.17. Purification section of gas blending manifold.